

PATENT SPECIFICATION

NO DRAWINGS

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COMPLETE SPECIFICATION

Preparation of a hydroxysebacic acid

We, GEIGY (U.K.) LIMITED, a British Company, whose Registered Office address is Simonsway, Manchester 22, Lancashire, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

The present invention relates to a process for the production of an hydroxy derivative of a dicarboxylic acid and in particular to a process for the production of 5-hydroxysebacic acid.

According to the present invention, a process for the production of 5-hydroxysebacic acid or an alkali metal salt thereof comprises contacting 6-hydroxy-1,10-decanedioic acid-1, 6-lactone with an aqueous medium comprising an alkali metal hydroxide.

6-Hydroxy-1,10-decanedioic acid-1,6-lactone and a process for the production thereof are described and claimed in our copending British patent application No. 21512/68 (Serial No. 1,196,595).

The process of the present invention is conveniently effected by contacting 6-hydroxy-1,10-decanedioic acid-1, 6-lactone with an aqueous solution of an alkali metal hydroxide, for instance sodium or potassium hydroxide, and heating the mixture under reflux conditions for a period of, for example, up to three hours. The concentration of the alkali metal hydroxide solution is advantageously within the range of from 10% to 40% by weight based on the total weight of the alkaline solution.

The 5-hydroxysebacic acid thus produced is in the form of the di-alkali metal salt which is however readily converted into the free acid form by rendering the reaction mixture acidic, for instance with concentrated hydrochloric acid. In this way, the 5-hydroxy-

sebacic acid may be precipitated from the reaction mixture and further purified, if desired, by conventional means such as fractional crystallisation.

The 5-hydroxysebacic acid produced according to the process of the present invention either in the form of the free acid or an alkali metal salt is particularly useful as an intermediate for the production of sebacic acid, especially in accordance with the process described and claimed in our copending British patent application No. 52602/66 (Serial No. 1,196,594).

The following Example further illustrates the present invention. Parts by weight expressed therein bear the same relation to parts by volume as do kilograms to litres.

Example Production of 6-hydroxy-1,10-decanedioic acid-1,6-lactone

As described in the Example in our copending application No. 21512/68 (Serial No. 1,196,595), 15.2 parts of 1,6-oxidocyclodec-1-ene dissolved in 100 parts by volume of methanol was treated at $-40^{\circ}\text{C}.$ with a stream of ozonised oxygen until the effluent gases gave a ready precipitate of iodine from an aqueous potassium iodide/boric acid solution. The methanol solvent was removed at $25^{\circ}\text{C}.$, under reduced pressure, and to the remaining oil there was added 70 parts by volume of 90% weight/weight formic acid and 20 parts by volume of 50% weight/weight hydrogen peroxide. This mixture was heated gently until a vigorous exothermic reaction set in. After the initial reaction had subsided, external heating at $100^{\circ}\text{C}.$, was continued for 15 minutes and the mixture was finally heated under reflux conditions for a further 30 minutes.

The water and the formic acid were removed from the reaction mixture under re-

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duced pressure. The oil remaining was then distilled under high vacuum and provided the following fractions:—

- (i) 2.7 parts of a liquid having a boiling range of up to 212°C. at 0.2 millimetres of mercury pressure,
- (ii) 13.1 parts of a liquid having a boiling point of 212°C. at 0.2 millimetres of mercury pressure, and
- (iii) 4.5 parts of a high boiling residue.

The main fraction boiling at 212°C. (0.2 millimetres) was partially crystalline in form and had an acid value of 309 milligrams of KOH per gram (6-hydroxy-1,10-decanedioic acid 1, 6-lactone has a theoretical acid value of 280 milligrams of KOH per gram).

Re-crystallisation of the crude partially-crystalline product from diethyl ether at 0°C. gave white prisms having melting-point of 46°C., an acid value of 281 milligrams of KOH per gram and the following elemental analysis by weight:—

	Found	Required (for $C_{10}H_{16}O$)
carbon	59.79	59.98
hydrogen	7.63	8.05

Production of 5-hydroxysebacic acid

To the oil produced above after removal of water and formic acid, were added 15 parts of potassium hydroxide dissolved in 50 parts by volume of water, and this mixture was heated under reflux conditions for a period of one hour. At the end of this time, the mixture was cooled and treated dropwise, with concentrated hydrochloric acid until the mixture acquired a pH value of 4.0. The white solid which separated on acidification was separated by filtration, washed with a little cold water and dried to produce 16.9 parts of 5-hydroxysebacic acid, having melting-point of 97° to 100°C., representing a yield of 77% theoretical.

WHAT WE CLAIM IS:—

1. A process of producing 5-hydroxysebacic acid or an alkali metal salt thereof comprising contacting 6-hydroxy-1,10-decanedioic acid-1,6-lactone with an aqueous medium comprising an alkali metal hydroxide.

2. A process as claimed in claim 1 wherein the alkali metal hydroxide is sodium or potassium hydroxide.

3. A process as claimed in claim 1 or 2 wherein the concentration of the aqueous alkali metal hydroxide solution is within the range of from 10% to 40% by weight based on the total weight of the alkaline solution.

4. A process as claimed in any of the preceding claims wherein the hydrolysis is carried out for up to three hours.

5. A process as claimed in any of the preceding claims wherein the hydrolysis is effected by heating the reaction mixture under reflux conditions.

6. A process as claimed in any of the preceding claims wherein 5-hydroxysebacic acid in the form of the free acid is obtained by rendering the reaction mixture acidic.

7. A process of producing 5-hydroxysebacic acid or an alkali metal salt thereof substantially as described in the Example.

8. 5-Hydroxysebacic acid or an alkali metal salt thereof whenever produced by a process claimed in any of the preceding claims.

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